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Standard Method of Test for
DENSITY OF PLASTICS BY THE DENSITY-GRADIENT TECHNIQUE[1]

This Standard is issued under the fixed designation D 1505; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval.

NOTE—Editorial change in Section 6.1 was made in September 1969.

1. Scope

1.1 This method covers determination of the density of solid plastics.

1.2 The method is based on observing the level to which a test specimen sinks in a liquid column exhibiting a density gradient, in comparison with standards of known density.

NOTE 1—The values stated in U.S. customary units are to be regarded as the standard. The metric equivalents of U.S. customary units may be approximate.

2. Significance

2.1 The density of a solid is a conveniently measurable property which is frequently useful as a means of following physical changes in a sample, as an indication of uniformity among samples, and a means of identification.

2.2 This method is designed to yield results accurate to better than 0.05 percent.

NOTE 2—Where accuracy of 0.05 percent or better is desired, the gradient tube shall be constructed so that vertical distances of 1 mm shall represent density differences no greater than 0.0001 g/cm.^{3} The sensitivity of the column is then 0.0001 g/cm^3 · mm. Where less accuracy is needed, the gradient tube shall be constructed to any required sensitivity.

3. Definition

3.1 *density of plastics*—the weight per unit volume of material at 23 C, expressed as follows:

$$D^{23C}, \text{g/cm}^3$$

NOTE 3—Density is to be distinguished from specific gravity, which is the ratio of the weight of a given volume of the material to that of an equal volume of water at a stated temperature.

4. Apparatus

4.1 *Density-Gradient Tube*—A suitable graduate with ground-glass stopper.[2]

4.2 *Constant-Temperature Bath*—A means of controlling the temperature of the liquid in the tube at 23 ± 0.1 C. A thermostatted water jacket around the tube is a satisfactory and convenient method of achieving this.

4.3 *Glass Floats*—A number of calibrated glass floats covering the density range to be studied and approximately evenly distributed throughout this range.

4.4 *Pycnometer*, for use in determining the densities of the standard floats.

4.5 *Liquids*, suitable for the preparation of a density gradient (Table 1).

NOTE 3—It is very important that none of the liquids used in the tube exert a solvent or chemical effect upon the test specimens during the time of specimen immersion.

4.6 *Hydrometers*—A set of suitable hydrometers covering the range of densities to be measured. These hydrometers should have 0.001 density graduations.

4.7 *Analytical Balance*, with a sensitivity of 0.001 g.

4.8 *Siphon or Pipet Arrangement*, for filling the gradient tube. This piece of equipment should be constructed so that the rate of flow of liquid may be regulated to 10 ± 5 ml/min.

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[1] This method is under the jurisdiction of ASTM Committee D-20 on Plastic and is the direct responsibility of Subcommittee D-20.70 on Analytical Methods.

Current edition effective Sept. 9, 1968. Originally issued 1957. Replaces D 1505 – 67.

[2] Tubes similar to those described in References (**6**) and (**12**) may also be used.

5. Test Specimen

5.1 The test specimen shall consist of a piece of the material under test. The piece may be cut to any shape convenient for easy identification, but should have dimensions that permit the most accurate position measurement of the center of volume of the suspended specimen (Note 5). Care should be taken in cutting specimens to avoid change in density resulting from compressive stress.

NOTE 5—The equilibrium positions of film specimens in the thickness range from 0.025 to 0.051 mm (0.001 to 0.002 in.) may be affected by interfacial tension. If this effect is suspected, films not less than 0.127 mm (0.005 in.) in thickness should be tested.

5.2 The specimen shall be free of foreign matter and voids and shall have no cavities or surface characteristics that will cause entrapment of bubbles.

6. Preparation of Density-Gradient Columns

6.1 *Preparation of Standard Glass Floats*[3]—Prepare glass floats by any convenient method such that they are fully annealed, approximately spherical, have a maximum diameter less than one quarter the inside diameter of the column and do not interfere with the test specimens. Prepare a solution (400 to 600 ml) of the liquids to be used in the gradient tube such that the density of the solution is approximately equal to the desired lowest density. When the floats are at room temperature, drop them gently into the solution. Save the floats that sink very slowly, and discard those that sink very fast or save them for another tube. If necessary to obtain a suitable range of floats, grind selected floats to the desired density by rubbing the head part of the float on a glass plate on which is spread a thin slurry of 400 or 500-mesh silicon carbide (Carborundum) or other appropriate abrasive. Progress may be followed by dropping the float in the test solution at intervals and noting its change in rate of sinking.

6.2 *Calibration of Standard Glass Floats* (See Appendix A1):

6.2.1 Place a tall cylinder in the constant-temperature bath maintained at 23 ± 0.1 C. Then fill the cylinder about two thirds full with a solution of two suitable liquids, the density of which can be varied over the desired range by the addition of either liquid to the mixture. After the cylinder and solution have attained temperature equilibrium, place the float in the solution, and if it sinks, add the denser liquid by suitable means with good stirring until the float reverses direction of movement. If the float rises, add the less dense liquid by suitable means with good stirring until the float reverses direction of movement.

6.2.2 When reversal of movement has been observed, reduce the amount of the liquid additions to that equivalent to 0.0001 g/cm^3 density. When an addition equivalent to 0.0001 g/cm^3 density causes a reversal of movement, or when the float remains completely stationary for at least 15 min, the float and liquid are in satisfactory balance. The cylinder must be covered whenever it is being observed for balance, and the liquid surface must be below the surface of the liquid in the constant-temperature bath. After vigorous stirring, the liquid may continue to move for a considerably length of time; make sure that the observed movement of the float is not due to liquid motion by waiting at least 15 min after stirring has stopped before observing the float.

6.2.3 When balance has been obtained, fill a freshly cleaned and dried pycnometer with the solution and place it in the 23 ± 0.1 C bath for sufficient time to allow temperature equilibrium of the glass. Determine the density of the solution by normal methods (ASTM Method D 941, Test for Density and Specific Gravity of Liquids by Lipkin Bicapillary Pycnometer[4]) and make "in vacuo" corrections for all weighings. Record this as the density of the float. Repeat the procedure for each float.

6.3 *Gradient Tube Preparation* (see Appendix for details):

6.3.1 *Method A*—Stepwise addition.

6.3.2 *Method B*—Continuous filling (liquid entering gradient tube becomes progressively less dense).

6.3.3 *Method C*—Continuous filling (liquid entering gradient tube becomes progressively more dense).

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[3] Glass floats may be purchased from the Scientific Glass Apparatus Co., Bloomfield, N. J.

[4] *Annual Book of ASTM Standards*, Part 17.

7. Conditioning

7.1 Test specimens whose change in density on conditioning may be greater than the accuracy required of the density determination shall be conditioned before testing in accordance with the method listed in the applicable ASTM material specification.

8. Procedure

8.1 Wet three representative test specimens with the less dense of the two liquids used in the tube and gently place them in the tube. Allow the tube and specimens to reach equilibrium, which will required 10 min or more. Thin films of 1 to 2 mils in thickness require approximately 1½ h to settle, and rechecking after several hours is advisable (Note 5).

8.2 When a graduated tube is used, read the height of the floats and specimens by using a line through their center of volume. When a cathetometer is used, measure the height of the floats and specimens from an arbitrary level using a line through their center of volume. If equilibrium is not obtained, the specimen may be imbibing the liquid.

8.3 Old samples can be removed without destroying the gradient by slowly withdrawing a wire screen basket attached to a long wire (Note 6). This can be conveniently done by means of a clock motor. Withdraw the basket from the bottom of the tube and, after cleaning, return it to the bottom of the tube. It is essential that this procedure be performed at a slow enough rate (approximately 30 min/300-mm length of column) so as not to disturb the density gradient.

NOTE 6—Whenever it is observed that air bubbles are collecting on samples in the column, a vacuum applied to the column will correct this.

9. Calculations

9.1 The densities of the samples may be determined graphically or by calculation from the levels to which the samples settle by either of the following methods:

9.1.1 *Graphical Calculation*—Plot float position versus float density on a chart large enough to be read accurately to ±1 mm and the desired precision of density. Plot the positions of the unknown specimens on the chart and read their corresponding densities.

9.1.2 *Numerical Calculation*—Calculate the density by interpolation as follows:

$$\text{Density at } x = a + [(x - y)(b - a)/(z - y)]$$

where:

a and b = densities of the two standard floats,

y and z = distances of the two standards, a and b, respectively, bracketing the unknown measured from an arbitrary level, and

x = distance of unknown above the same arbitrary level.

10. Report

10.1 The report shall include the following:

10.1.1 Density reported as D^{23C}, in grams per cubic centimeter, as the average for three representative test specimens,

10.1.2 Number of specimens tested if different than three,

10.1.3 Sensitivity of density gradient in grams per cubic centimeter per millimeter,

10.1.4 Complete identification of the material tested, and

10.1.5 Date of the test.

REFERENCES

(1) Linderstrøm-Lang, K., "Dilatometric Ultra-Micro-Estimation of Peptidase Activity," *Nature*, NATRA, Vol 139, 1937, p. 713.

(2) Linderstrøm-Lang, K., and Lanz, H., "Enzymic Histochemistry XXIX Dilatometric Micro-Determination of Peptidase Activity," *Comptes rendus des gravaus de laboratorie Carlsberg*, *Serie Chimique*, Vol 21, 1938, p. 315.

(3) Linderstrøm-Lang, K., Jacobsen, O., and Johansen, G., "Measurement of the Deuterium Content in Mixtures of H_2O and D_2O," *ibid.*, Vol 23, 1938, p. 17.

(4) Jacobsen, C. F., and Linderstrøm-Lang, K., "Method for Rapid Determination of Specific Gravity," *Acta Physiologica Scandinavica*, APSCA, Vol 1, 1940, p. 149.

(5) Boyer, R. F., Spencer, R. S., and Wiley, R. M., "Use of Density-Gradient Tube in the Study of High Polymers," *Journal of Polymer Science*, JPSCA, Vol 1, 1946, p. 249.

(6) Anfinsen, C., "Preparation and Measurement

of Isotopic Tracers: A Symposium Prepared for the Isotope Research Group," Edwards, J. W., Publishers, Ann Arbor, Mich., 1946, p. 61.

(7) Tessler, S., Woodberry, N. T., and Mark H., "Application of the Density-Gradient Tube in Fiber Research," *Journal of Polymer Science*, JPSCA, Vol 1, 1946, p. 437.

(8) Low, B. W., and Richards, F. M., "The Use of the Gradient Tube for the Determination of Crystal Densities," *Journal of the American Chemical Society*, JACSA, Vol 74, 1952, p. 1660.

(9) Sperati, C. A., Franta, W. A., and Starkweather, H. W., Jr., "The Molecular Structure of Polyethylene V, the Effect of Chain Branching and Molecular Weight on Physical Properties," *Journal of the American Chemical Society*, JACSA, Vol 75, 1953, p. 6127.

(10) Tung, L. H., and Taylor, W. C., "An Improved Method of Preparing Density Gradient Tubes," *Journal of Polymer Science*, JPSCA, Vol 21, 1956, p. 144.

(11) Mills, J. M., "A Rapid Method of Construction Linear Density Gradient Columns," *Journal of Polymer Science*, Vol 19, 1956, p. 585.

(12) Wiley, R. E., "Setting Up a Density Gradient Laboratory," *Plastics Technology*, PLTEA, Vol 8, No. 3, 1962, p. 31.

TABLE 1 Liquid Systems for Density-Gradient Tubes

System	Density Range, g/cm^3
Methanol - benzyl alcohol	0.80 to 0.92
Isopropanol - water	0.79 to 1.00
Isopropanol - diethylene glycol	0.79 to 1.11
Ethanol - carbon tetrachloride	0.79 to 1.59
Toluene - carbon tetrachloride	0.87 to 1.59
Water - sodium bromide	1.00 to 1.41
Water - calcium nitrate	1.00 to 1.60
Carbon tetrachloride - trimethylene dibromide	1.60 to 1.99
Trimethylene dibromide - ethylene bromide	1.99 to 2.18
Ethylene bromide - bromoform	2.18 to 2.89

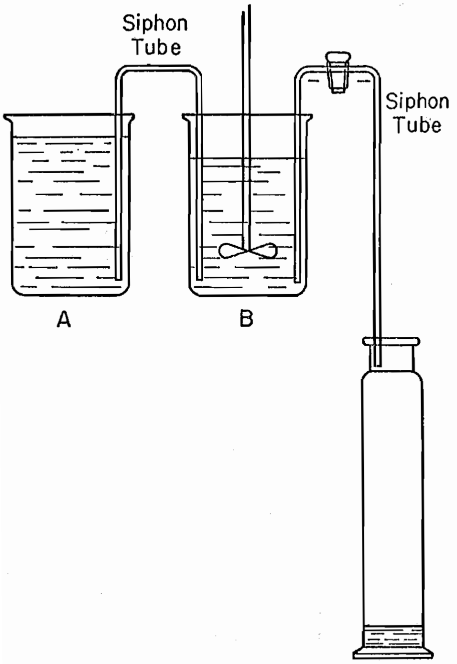

FIG. 1 Apparatus for Gradient Tube Preparation (Method B).

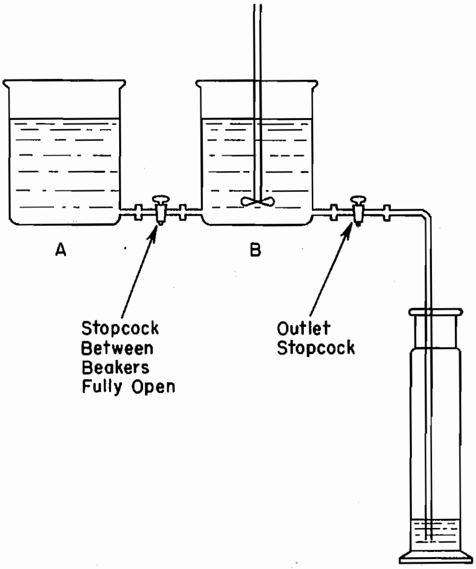

FIG. 2 Apparatus for Gradient Tube Preparation (Method C).

APPENDIXES

A1. FLOAT CALIBRATION—ALTERNATIVE METHOD

A1.1 This method of float calibration has been found by one laboratory to save time and give the same accuracy as the standard method. Its reliability has not been demonstrated by round-robin data.

A1.1.1 Prepare a homogeneous solution whose density is fairly close to that of the float in question.

A1.1.2 Fill a graduate about $^3/_4$ full with the solution, drop in the float, stopper, and place in a thermostatted water bath near 23 C. Fill a tared two-arm pycnometer (ASTM Method D 941,[4] or equivalent) with the solution. Place the pycnometer in the bath.

A1.1.3 Vary the bath temperature until the solution density is very near to that of the float. (If the float was initially on the bottom of the graduate, lower the bath temperature until the float rises; if the float floated initially, raise the bath temperature until the float sinks to the bottom.)

A1.1.4 Change the bath temperature in the appropriate direction in increments corresponding to solution density increments of about 0.0001 g/cm^3 until the float reverses direction of movement as a result of the last change. This must be done slowly (at least 15-min intervals between incremental changes on the temperature controller). Read the volume of liquid in the pycnometer.

A1.1.5 Change the bath temperature in increments in the opposite direction, as above, until a change in the float position again occurs. Read the volume of liquid in the pycnometer.

NOTE A1—The float should rise off the bottom of its own volition. As a precaution against surface tension effects when the float is floating, the float should be pushed about half-way down in the liquid column and then observed as to whether it rises or falls. For this purpose, a length of Nichrome wire, with a small loop on the lower end and an inch or so of length extending above the liquid surface, is kept within the graduate throughout the course of the run. To push a floating float down, the cylinder is unstoppered and the upper wire end grasped with tweezers for the manipulations. The cylinder is then quickly restoppered.

A1.1.6 Remove the pycnometer from the bath, dry the outside, and set aside until the temperature reaches ambient temperature. Weigh and calculate the "in vacuo" mass of solution to 0.0001 g. Using the average of the two observed solution volumes, calculate the density of the solution to 0.0001 g/cm^3. This solution density is also the float density.

A1.1.7 The pycnometer used should be calibrated for volume from the 23 C calibration, although the reading is taken at a different temperature. The alternative method is based on a number of unsupported assumptions but generally gives the same results as that described in 6.2 within the accuracy required. In case of disagreement, the method described in 6.2 shall be the referee method.

A2. GRADIENT TUBE PREPARATION

A2.1 *Method A—Stepwise Addition:*

A2.1.1 Using the two liquids that will give the desired density range, and sensitivity (S) in grams per cubic centimeter per millimeter, prepare four or more solutions such that each differs from the next heavier by 80 S g/cm^3. The number of solutions will depend upon the desired density range of the column and shall be determined as follows:

$$\text{Number of solutions to prepare density-gradient column (Note A2)} = (1 + D_2 - D_1)/80\,S$$

where:

D_2 = upper limit of density range desired,
D_1 = lower limit of density range desired, and
S = sensitivity, in grams per cubic centimeter per millimeter.

NOTE A2—Correct the value of $(1 + D_2 - D_1)/80\,S$ to the nearest whole number. To prepare these solutions, proceed as follows: Using the hydrometers, mix the two liquids in the proportions necessary to obtain the desired solutions. Remove the dissolved air from the solutions by gentle heating or an applied vacuum. Then check the density of the solutions at 23 ± 0.1 C by means of the hydrometers and, if necessary, add the appropriate air-free liquid until the desired density is obtained.

NOTE A3—Where aqueous mixtures are used, 0.5 percent aqueous sodium acetate should be used to prepare the mixture. This reduces the formation of bubbles from dissolution.

NOTE A4—In order to obtain a linear gradient in the tube, it is very important that the solutions be homogeneous and at the same temperature when their densities are determined. It is also important that the density difference between the solutions consecutively introduced into the tube be equal.

A2.1.2 By means of a siphon or pipet, fill the gradient tube with an equal volume of each liquid starting with the heaviest, taking appropriate measures to prevent air from being dissolved in the liquid. After the addition of the heaviest liquid, very carefully and slowly pour an equal volume of the second heaviest liquid down the side of the column by holding the siphon or pipet against the side of the tube at a slight angle. Avoid excess agitation and turbulence. In this manner, the "building" of the tube shall be completed.

NOTE A5—Density gradients may also be prepared by reversing the procedure described in A2.1.1 and A2.1.2. When this procedure is used, the lightest solution is placed in the tube and the next lightest solution is very carefully and slowly "placed" in the bottom of the tube by means of a pipet or siphon which just touches the bottom of the tube. In this manner the "building" of the tube shall be completed.

A2.1.3 If the tube is not already in a constant-temperature bath, transfer the tube, with as little agitation as possible, to the constant-temperature bath maintained at 23 ± 0.1 C. The bath level should approximately equal that of the solution in the tube, and provision should be made for vibrationless mounting of the tube.

A2.1.4 For every 254 mm of length of tube, dip a minimum of five clean calibrated floats, spanning the effective range of the column, into the less dense solvent used in the preparation of the gradient tube and add them to the tube. By means of a stirrer (for example, a small coiled wire or other appropriate stirring device) mix the different layers of the tube gently by stirring horizontally until the least dense and most dense floats span the required range of the gradient tube. If at this time it is observed that the floats are "bunched" together and not spread out evenly in the tube, discard the solution and repeat the procedure. Then cap the tube and keep it in the constant-temperature bath for a minimum of 24 h.

A2.1.5 At the end of this time, plot the density of floats versus the height of floats to observe whether or not a fairly smooth and nearly linear curve is obtained. Some small irregularities may be seen, but they should be slight. Whenever an irregular curve is obtained, the solution in the tube shall be discarded and a new gradient prepared.

NOTE A6—Gradient systems may remain stable for several months.

A2.2 *Method B—Continuous Filling with Liquid Entering Gradient Tube Becoming Progressively Less Dense:*

A2.2.1 Assemble the apparatus as shown in Fig. 1, using beakers of the same diameter. Then select an appropriate amount of two suitable liquids which previously have been carefully deaerated by gentle heating or an applied vacuum. Typical liquid systems for density-gradient tubes are listed in Table 1. The volume of the more dense liquid used in the mixer (beaker *B* shown in Fig. 1) must be equal to at least one half of the total volume desired in the gradient tube. An estimate of the volume of the less dense liquid required in beaker *A* to establish flow from *A* to *B* can be obtained from the following inequality:

$$V_A > d_B V_B / d_A$$

where:

V_A = starting liquid volume in beaker *A*,
V_B = starting liquid volume in beaker *B*,
d_A = density of the starting liquid in beaker *A*, and
d_B = density of the starting liquid in beaker *B*.

A small excess (not exceeding 5 percent) over the amount indicated by the above equality will induce the required flow from *A* to *B* and yield a very nearly linear gradient column.

A2.2.2 Place an appropriate volume of the denser liquid into beaker *B* of suitable size. Prime the siphon between beaker *B* and the gradient tube with liquid from beaker *B* and then close the stopcock. The delivery end of this siphon should be equipped with a capillary tip for flow control.

A2.2.3 Place an appropriate volume of the less dense liquid into beaker *A*. Prime the siphon between beakers *A* and *B* with the liquid from beaker *A* and close the stopcock. Start the highspeed, propeller-type stirrer in beaker *B* and adjust the speed of stirring such that the surface of the liquid does not fluctuate greatly.

A2.2.4 Start the delivery of the liquid to the gradient tube by opening the two siphon-tube stopcocks simultaneously. Adjust the flow of liquid into the gradient tube at a very slow rate, permitting the liquid to flow down the side of the tube. Fill the tube to the desired level.

NOTE A7—Preparation of a suitable gradient tube may require 1 to 1½ h or longer, depending upon the volume required in the gradient tube.

A2.3 *Method C—Continuous Filling with Liquid Entering Gradient Tube Becoming Progressively More Dense:*

A2.3.1 This method is essentially the same as Method B with the following exceptions:

A2.3.2 The lighter of the two liquids is placed in beaker *B*.

A2.3.3 The liquid introduced into the gradient column is introduced at the bottom of the column. The first liquid introduced is the lighter end of the gradient and is constantly pushed up in the tube as the liquid being introduced becomes progressively heavier.

A2.3.4 The liquid from beaker *A* must be introduced into beaker *B* by direct flow from the bottom of beaker *A* to the bottom of beaker *B*, rather than being siphoned over as it is in Method B. Filling the tube by this method may be done more rapidly than by Methods A or B. The stopcock between containers *A* and *B* should be of equal or larger bore than the outlet stopcock. A schematic drawing of the apparatus for Method C is shown in Fig. 2.